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| L10 | fr-2787322\$.did.                                      | 2    | L10 |
| L9  | L8 and amphipathic                                     | 66   | L9  |
| L8  | moisturizer and (glycerol or glycerin) and surfactant  | 2193 | L8  |
| L7  | L6 and amphipathic                                     | 56   | L7  |
| L6  | moisturizer and (glycerol or glycerine) and surfactant | 1871 | L6  |

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| L5 | WO-9406400\$.did.                                      | 1    | L5 |
| L4 | moisturizer and (glycerol or glycerine) and surfactant | 1860 | L4 |

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|----|---|---|----|
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| L2 | moisturizer and (glycerol or glycerine) and amphipathic         | 0 | L2 |
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## Formulation of conditioning shampoos

*Marianne D Berthiaume*. [DCI](#). New York: [May 1997](#). Vol. 160, Iss. 5; pg. 54, 7 pgs>> [Jump to full text](#)

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### Abstract (Article Summary)

The base constituents of a shampoo formulation are water and a combination of surfactants, typically anionic detergents. The surfactants act to emulsify the accumulated surface oils, allowing their removal during the rinsing process. Specialty shampoos may contain conditioning agents, materials to protect or refresh hair color, sunscreens, aromatherapeutic agents, etc. The various classes of ingredients typically utilized in formulation of conditioning shampoo products are discussed. Examples of conditioning shampoo formulas are presented.

### Full Text (3084 words)

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In formulating a shampoo, the primary objective is to cleanse the hair and scalp of hydrophobic oil and soil, and therefore the base constituents of a shampoo formulation are water and a combination of surfactants, typically anionic detergents. The surfactants act to emulsify the accumulated surface oils, allowing their removal during the rinsing process. Ideally, a shampoo should exhibit gentle cleansing effects, good flash foam, and good rinsability, and the product must be nontoxic and non-irritating to the skin and eyes. Specialty shampoos may include conditioning agents, dandruff control agents, materials to protect or "refresh" hair color, sunscreens, aromatherapeutic agents, etc. To this end, the base composition contains, in addition to the surfactants, materials such as foam boosters and stabilizers, viscosity control agents, preservatives, colors, fragrances, opacifying agents, vitamins, sunscreens, botanical extracts, and conditioning agents such as cationic surfactants or **polymers**, or more commonly, silicones. The following discussion details the various classes of ingredients typically utilized in formulation of conditioning shampoo products, providing examples for each category.

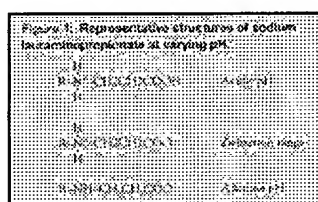
Surfactants are "amphipathic" molecules containing a hydrophilic head group and a hydrophobic tail. These compounds are surface active agents which reduce the surface tension of water and lower the interfacial tension between two immiscible substances, e.g. sebum and water. The primary purpose of incorporating surfactants into

shampoo products is as cleansing agents; however, they may also be utilized as emulsifying agents, hydrotropes, suspending agents, thickeners, solubilizers, or foam boosters, depending on the surface active agent of choice. Surfactants may be classified as anionic, nonionic, cationic, or amphoteric depending on their specific components. Anionic surfactants are generally utilized as the primary cleansing agent in shampoo systems, and except for water are the major component in the product. Anionic surfactants are very effective detergents, and while they may be well suited for "clarifying" products, their deterative action may be too harsh in other systems, completely stripping the hair of its natural oils and lipids, and leaving the hair dull and brittle. Because their deterative properties may be easily altered by the addition of secondary surfactants, and because of their low cost alkyl sulfates and alkyl ether sulfates are the most commonly used anionic surfactants employed in shampoo products. Anionic surfactants are generally added to the formulation in the range of 10-25 percent.

Nonionic surfactants are generally used as the secondary surfactants or co-surfactants in a shampoo product. Nonionic surfactants tend to enhance the foam quality, increasing both density and stability, and by forming mixed micelles with the primary surfactants, they act to alter the viscosity of the product. Particularly useful in this application are the alkanolamides and amine oxides which form hydrogen bonds through the amine cation and the surfactant anion resulting in a closer packed film at the liquid-air interface. This reduces foam collapse caused by gas diffusion between two bubbles in contact, and the foam is generally characterized as being "richer" or "creamier," having smaller, more uniform bubbles. Other nonionic surfactants, especially the ethoxylated alcohols, are employed as solubilizers or wetting agents, while nonionic silicone glycol copolymers reduce eye irritation and provide light conditioning benefits. Nonionic surfactants may also act as opacifying agents in shampoos. Typical usage levels of nonionics in a shampoo composition are a quarter of the level of the anionic surfactant contained in the composition.

Amphoteric surfactants possess groups which may be positively or negatively charged, or neutral, depending on the solution pH. This property is illustrated in Figure 1, which depicts the structure of sodium lauraminopropionate under acidic, neutral and basic conditions.

When an amphoteric surfactant is in the zwitterion range it possesses both positive and negative charges, and is referred to as being an "inner salt." In this pH range, the compound has no surfactant properties and may actually interfere with foam production. An amphoteric surfactant may, however, be used to help stabilize foam under the proper conditions, and these materials are frequently employed as viscosity enhancing agents for their ability to form mixed micelles with the primary surfactants, driving the transition from spherical to cylindrical structures. Amphoteric surfactants are known for their mildness, and are often utilized in the baby shampoo formulations. They may be incorporated into shampoo formulations in levels ranging from 2-20 percent depending on the other surfactants employed in the formulation.



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Figure 1:

Cationic surfactants are generally not used in shampoos as part of the cleansing system. They are not particularly effective detergents and they are incompatible with the anionic primary surfactants. Cationic surfactants are often used in conditioning shampoos, however, for their ability to impart softness and antistatic properties to the dry hair, to increase or alter the zeta potential of other conditioning actives, and to increase the deposition of unmodified silicones onto hair. Cationic surfactants are generally added to conditioning shampoo formulations in levels of 0.5-3.0% active.

Conditioning agents are added to shampoos to make the hair easier to comb, to reduce fly away, and to make the dry hair soft, manageable and lustrous. These materials may include super-fattening agents, silicones, humectants, cationic surfactants or **polymers**, hydrocarbon oils or waxes, proteins and lipids. Super-fattening agents may include fatty esters, natural oils, ethoxylated triglycerides and lanolin derivatives. Conditioning agents are typically used at levels of 0.5-10% to impart softness, luster and manageability to the hair without reducing the foam of the system. Humectants include materials such as

**glycerin** or propylene glycol and are claimed to add moisture to the hair, reducing the generation of static charge and increasing fiber elasticity. They are frequently incorporated into the formulation to act as clarifying agents, matching the refractive indices between other ingredients, and to help retard water loss from the product. Typical usage levels are 1-10 percent. Cationic **polymers**, proteins and amino acids are very efficacious conditioning agents due to their substantivity to hair. Some studies suggest that amino acids will actually penetrate into the hair and increase the moisture content as well. Proteins and cationic **polymers** typically remain on the fiber surface, reducing combing forces and fly-away, and in some systems, they provide enhancement of volume and body and an improvement in manageability. These ingredients may be found in shampoo formulations in concentrations ranging from 0.5-10.0%. Silicones may be incorporated into shampoos in this same concentration range to reduce eye irritation, combing forces, static charge, and drying times while increasing foam stability, body, shine and manageability depending on the particular silicone materials chosen. Table I lists the benefits derived from incorporation of some common silicone compounds in shampoo formulations.

Opacifying agents and pearling agents are added to specifically reduce the clarity, improve aesthetics, and occasionally to give the impression of a more sophisticated or complex system designed to provide some additional benefits. These materials may include fatty alcohols, copolymers of styrene or acrylates, inorganic clays, minerals, polyol esters, etc. The typical usage levels range from 0.2-5.0%. Opacifiers may cause a decrease in foam production while yielding a denser foam. The most common pearling agent encountered in shampoos is ethylene glycol distearate. Pearls produced by these materials are often referred to as "pourable pearls," meaning that the composition may appear flat upon standing but will provide a pearlized appearance during pouring. This phenomenon results from formation of platelets by the distearate during processing, which undergo uniaxial alignment in order to decrease internal friction under flow conditions. The pearlescence obtained with distearates depends largely on the size, shape, distribution and reflectance of the crystals formed during the cooling process, which in turn may be related to the salt content of the system, the mono- to di-ester ratio, and the particular cut of alkyl chain length. In addition to acting as opacifiers, glycol and **glycerol** stearates also affect the viscosity of the product. Inorganic pearls such as micronized titanium dioxide are not affected by rate of cooling during production, but these materials require a more viscous base, and usually necessitate addition of suspending agents, in order to avoid settling of the pearl under gravity, particularly at elevated temperatures.

Viscosity controlling agents may be added to the formulation to increase or decrease viscosity. The simplest example of this phenomenon is observed with the addition of salt (typically chlorides of sodium, ammonium or magnesium) as illustrated in Figure 2. As one begins to add salt to the composition the viscosity increases as the micellar shape changes from spherical to cylindrical. Beyond a certain point (which varies for different formulations), the viscosity rapidly and irreversibly decreases. This process is often referred to as a "salting out" effect. When thickening by addition of salt, it is advisable to select the proper salt concentration on the ascending portion of the curve so that trace impurities such as salt contaminants in surfactants do not push the composition into the low viscosity portion of the curve.

The process of thickening with electrolytes works best when employing standard anionic surfactants. When using alternative primary surfactants such as sarcosinates or sulfosuccinates, thickening by electrolyte addition may not be possible. In these systems, other types of thickening agents such as those described below may be required.

Acrylic acid **polymers** may act as suspending agents (particularly good for silicones) as well as thickeners. They are difficult to disperse, require neutralization, and generally display poor electrolyte tolerance. Cellulosic ethers display better electrolyte tolerance, maintain viscosity at elevated temperatures, and act as foam stabilizers. These materials are thickeners only and do not function as

suspending agents. They have the added disadvantage of being sticky or tacky at high concentrations, display pituitous rheology characteristics, and they are particularly susceptible to microbial attack. Natural gums or polysaccharides are characterized by good pH and electrolyte tolerance. As with the cellulosic ethers, natural gums are prone to microbial attack and tend to display pituitous rheology. Depending on the particular gum, they may act as suspending agents as well as thickeners (e.g., xanthan is known to be a good suspending agent for silicones in shampoos). The gums generally display good freeze/thaw characteristics, but may have inconsistent viscosity performance. Fatty alcohols thicken via insolubility properties - they are soluble inside the micelle, and therefore change the micellar structure. Inorganic clays are good thickening agents for compositions containing cyclomethicones, and are efficacious suspending agents for antidandruff actives, but not silicones.

Another method of thickening involves the use of newer high molecular weight thickeners which form association complexes in solution. Materials such as PEG-120 methylglucose dioleate, PEG-6000 distearate and talloweth-60 myristyl glycol are among these types of agents. A potential problem encountered with these types of thickeners is a change in flow properties leading to increased Newtonian flow as compared with saltthickened systems which exhibit pseudoplastic flow behavior. This alteration in rheology may lead to pituitive effects or "stringing" as opposed to providing a clean break of the droplets. External thickeners are usually added to the shampoo formulation in the range of 0.25-2.0% active.

Viscosity reducing agents include salt as mentioned above, hydrotropes, coupling agents which increase the solubility of one ingredient in another, low molecular weight silicones (particularly volatile silicones) and acids or bases. Since viscosity may be dependent on pH, shampoo formulations should be brought to the final pH range prior to any final viscosity evaluations. In general, viscosity increases as pH decreases except for soap based systems. Another method of reducing viscosity is to add a small amount to ethanol or isopropanol. These materials will act to reduce foam as well as viscosity, so their use must be limited.

**Foam Boosters/Stabilizers.** A foam booster is a material which will increase the amount of foam produced by a surfactant system at a constant molar concentration of surfactant -- obviously adding more surfactant will result in more foam up to a point, generally just above the critical micelle concentration, but this is not an acceptable method of increasing foam generation. Compounds which act to lower the CMC of the primary surfactant or to decrease the surface tension at the liquid/air interface will result in greater amounts of foam generation. Amine oxides, alkanolamines, betaines, and sultaines are known to be effective foam boosters in shampoo formulations. These materials may also impart some foam stabilization to most surfactant systems. A foam stabilizer is a material which will delay collapse of the foam, usually by decreasing the rate of liquid drainage, or increasing the viscoelastic film strength in the liquid film comprising the foam lamellae. Materials such as silicone glycol copolymers, natural gums, cellulosic derivatives, and small amounts of fatty alcohols will act as foam stabilizers. Unlike the foam boosters listed above which also provide foam stabilization, these substances will not increase foam generation. Foam boosters and/or stabilizers are typically added to a shampoo formulation in a range of from 1-5 percent, generally not exceeding 30 percent of the active surfactant concentration.

Table 1. Benefits derived from incorporation of various types of silicones in shampoo products

Sequestering agents are particular ligands or chelating agents which form coordinate bonds with metal

ions to produce water soluble complexes. Sequestrants are added to a shampoo composition to prevent deposition of insoluble calcium or magnesium soaps or other salts on the hair during rinsing - a common problem encountered when using hard water. They are also used to help avoid discoloration of the product and prevent rancidity (i.e. oxidation) of oils and other susceptible ingredients. Sequestering agents offer the additional benefit of improving effectiveness of some preservative systems, especially against *Pseudomonas* bacteria. Generally, 0.1-0.2% of the various ethylenediamino tetraacetic acid salts are used for this purpose. Citric acid and sodium citrate, in the same concentration range, may also provide sequestering benefits, as well as buffering the system.

Therapeutic agents, specifically for control of dandruff, seborrheic dermatitis and psoriasis are employed at concentrations of 0.5-5.0%. These materials are considered to be Category I Drugs, i.e. safe and effective. According to the FDA in 51 Fed. Reg. 27346, July 30, 1986, the following ingredients, at the specified levels, are the only approved compounds that can be used for treatment of the above listed conditions: coal tar (23.75%), pyrithione zinc (1-2%), salicylic acid (1.83.0%), and sulfur 2-5% (with selenium sulfide, SeS<sub>2</sub>, used at 1% levels).

Preservatives are essentially believed to be ingredients which are intended to prevent or retard microbial growth -- including bacteria, yeast, mold, virus and fungus. Typical usage levels are from 0.1-0.5%. Frequently, several microbial preservatives are used in combination to provide a broader spectrum of coverage, and to reduce irritation caused by these ingredients. In the strictest sense, preservatives include any materials added to a composition to maintain the quality, integrity, or safety of the product, or prevent adulteration of the formulation. In this function other types of ingredients not generally considered to be preservatives would fall into this category. One example would be sequestering agents which increase stability of the formulation by binding any metal ions, such as trace amounts of iron picked up from processing equipment, which would form insoluble complexes in the product. Other examples would include antioxidants and sunscreens which prevent discoloration and rancidity of natural oils, colors, and fragrance oils. Typical use levels for these materials is up to 2% of the oil phase.

Buffering agents or pH adjusters are added to shampoo formulations in order to maintain a constant product pH during storage and usage. These materials are particularly important when formulating with alkyl ether sulfates which are ester compounds and are therefore susceptible to hydrolysis reactions under low pH conditions (typically under 4). Buffers are also employed to keep the pH from drifting up with time, this action being particularly important when employing surfactants of ammonium salts which liberate ammonia at elevated pH values (typically above 7.5). Citric acid/sodium citrate is a common buffering system employed in shampoo formulations. This system, as mentioned above, will also act to bind free metal ions in the formulation preventing formation of insoluble complexes.



Figure 2. Representative salt curve depicting the relationship between viscosity and salt concentration in a shampoo system

Specialty additives such as colors, fragrances, proteins, amino acids, vitamins, botanical extracts and the like are typically added to the formulation after it has cooled to a temperature of 40degC or below.

## Product Evaluations

Stability evaluations should be run in glass as well as in the actual packaging. Package testing includes

label and ink, cap liners (glue and microbial attack), torque testing at 25deg and 50degC, and weight loss at various torques. Thermal stability is generally run at 45deg and 50degC for 3 months, at 38degC for 6 months, at 25deg, 4deg, and -10deg for one year, and freeze/thaw from 10deg to +25degC for 10 cycles. While running stability evaluations, the following criteria are monitored: particle size, viscosity -- should have at least 10% of ambient viscosity at 45degC, pH - downward drifting may cause hydrolysis of ester type surfactants, upward drifting may cause liberation of ammonia. Other parameters which must be monitored include preservative action -- parabens and phenoxyethanol may be absorbed by certain types of packaging material color, fragrance, and actives assays. The final performance test is to ship a case of product from the northeast to the southwest and back in both summer and winter.

Performance evaluations may include: combing measurements, hair volume analysis, triboelectric charging or fly-away, tensile properties of hair treated with particular formulations, half-head subjective analysis, shine evaluation, curl retention, foam generation and subsequent collapse, deposition, and toxicity testing including ocular, dermal, LD <sup>50</sup> and sensitization. Analysis of foam production and collapse may be run in the presence and absence of synthetic sebum.

### Example Formulations

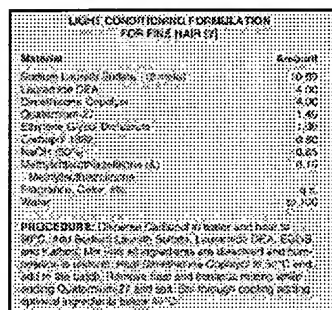
The following are several examples of conditioning shampoo formulations collected from the literature. Some of these compositions provide additional benefits such as clarity or dandruff control.



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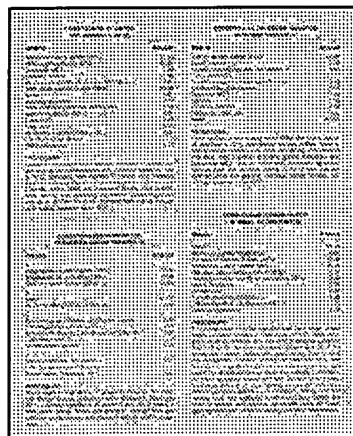
### CLEAR CONDITIONING SHAMPOO (GE SILICONES FORMULARY GUIDE)



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### LIGHT CONDITIONING FORMULATION FOR FINE HAIR [2]


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CONDITIONING SHAMPOO FOR NORMAL HAIR [4]

CONDITIONING DANDRUFF SHAMPOO WITH DIMETHICONE [4]

CONDITIONING SHAMPOO FOR PERMED OR COLOR TREATED [6]

CONDITIONING SHAMPOO FOR NORMAL TO DRY HAIR [3]


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1) CONDITIONING SHAMPOO FOR NORMAL TO OILY HAIR [1]

6) CONDITIONING SHAMPOO FOR CHILDREN [5]

### [Reference]

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